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# X-RAY TOPOGRAPHY UTILIZING NON-EQUATORIAL REFLECTIONS

P.W. KINGMAN

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## US ARMY BALLISTIC RESEARCH LABORATORY
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A technique for back-reflection topography has been developed which uses non-equatorial reflections. The geometry of back-reflection topography is analyzed in a general way, and it is shown how this analysis can be used for systematic imaging and contrast strategies. A novel camera design based upon this approach is also presented.

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# I. INTRODUCTION

The basic techniques of back reflection X-ray diffraction topography are well known and have been extensively described in the literature[1-5]. Since the early work of Berg[6] and Barrett[7] numerous investigators have devised a variety of specialized techniques for a wide range of applications. The overwhelming majority of these methods have implicitly assumed equatorial or near-equatorial geometry. This in turn imposes severe requirements on the relative orientation of the diffracting plane and the physical specimen surface. During the course of several investigations at BRL utilizing X-ray topography to study the deformation structure of metal crystals, a technique has been evolved which routinely utilizes non-equatorial reflections.

Equatorial geometry has generally been preferred, particularly in the study of deformation structures, because of the image distortion which is inherent in X-ray topography. For equatorial reflections, the distortion is a one-dimensional stretch. In all other cases the distortion becomes much more complex. In an earlier paper[8] it was shown that the general image distortion in back reflection topography can be described by an affine transformation. By appropriate choice of benchmarks, the components of the transformation can be determined; it is then possible to measure the coordinates of any feature on the topograph, apply a matrix transformation, and derive the true geometry. This opened up the possiblity of obtaining quantitative information about the geometry of deformation structures from non-equatorial topographs. In addition, a new camera was designed which allowed much greater flexibility in setting diffraction geometry. While this method evolved originally from the necessity of working with observation surfaces of arbitrary crystallographic orientation, as new equipment and working procedures developed it became evident that a much more powerful approach to classical back-reflection topography was evolving.

The main features of the BRL/Berg-Barrett method to be described are the following:

1. The design and implementation of a simple back-reflection camera which allows $360^\circ$ azimuthal rotation of the sample. This places every physically possible reflecting condition within reach with a single sample mounting.

2. To fully exploit this new camera, a method of stereographic analysis was developed in which the entire range of reflecting possibilities is systematically examined and an appropriate imaging or contrast strategy is selected in advance.

3. After every topograph, a double exposure check allows absolute identification of the diffracting conditions, as well as the detection of any unexpected strays or overlaps.

The BRL camera design, while mechanically simple, represents a departure from traditional simple back reflection designs and enormously enhances the power and flexibility of the Berg-Barrett and related techniques without requiring elaborate apparatus.

The routine use of a complete stereographic analysis for planning the imaging strategy, and the verification of the actual reflection conditions as standard procedure, have proven absolut°ly essential for meaningful results. Random searching for "good" images and rough guesses about probable Bragg reflections produce topographs which cannot be interpreted in any systematic way. Only when diffraction geometry is systematically selected and verified can contrast experiments be performed or the geometry of observed features be analyzed. Also, when dealing with deformed crystals, it is essential to eliminate the possibility of overlaps and ghosts.

The experimental techniques which have been developed will now be described.

## II. THEORETICAL ANALYSIS

The basic geometry for back-reflection X-ray topography has been extensively discussed by Barrett[7], Newkirk[2], and Honeycombe[4]. Equatorial geometry has generally been desired because image distortion is minimized, but is possible only if the pole of the diffracting plane is within $\theta$ degrees of the specimen surface normal, (where $\theta$ is the Bragg angle). (Many other reflections can occur, but they cannot be brought to the equatorial plane.) Once the sample is set with the pole of the diffracting plane lying in the plane defined by the direct beam and the specimen surface normal, the sample is tilted about an axis normal to this plane to achieve the Bragg angle.

A serious limitation in the design of many simple goniometers for X-ray topography is that the principal adjustment is the sample tilt, with only a very limited provision for other adjustments. If a single topograph using an equatorial setting is desired, the sample is initially mounted in this setting with only small final adjustments required. If several reflections and, in particular, non-equatorial geometries are used, however, the provision of $360°$ azimuth adjustment is of critical importance, since it permits not only a succession of settings for various reflections without remounting, but also the manipulation of diffracting conditions for contrast experiments and the elimination of troublesome overlapping images -- a particularly annoying problem with severely deformed crystals, where diffraction may occur over a range of several degrees. It also permits manipulation of azimuth and tilt to minimize the final tilt adjustment so as to achieve grazing incidence and maximize the irradiated area. The possibilities of non-equatorial geometry are best illustrated with the aid of Figures 1 through 3.

Figure 1 is a stereogram of a construction similar to that originally proposed by Leber[2]. It shows the loci of possible near-glancing angle reflections for a copper crystal with copper radiation. The interpretation is as follows: With the X-ray beam coming from the right, a (111) pole which is $90-\theta°$ from the beam (i.e., anywhere on the small circle marked (111) which is $90-\theta$ from the incoming beam) will produce a reflection lying at $2\theta$ from the exit beam (i.e., somewhere on the small circle of $2\theta$ about the exit beam). In the original construction, the only sample adjustment allowed was rotation about the axis normal to the beam ("TILT"); hence the requirement for obtaining a (111) reflection would be to have a (111) pole

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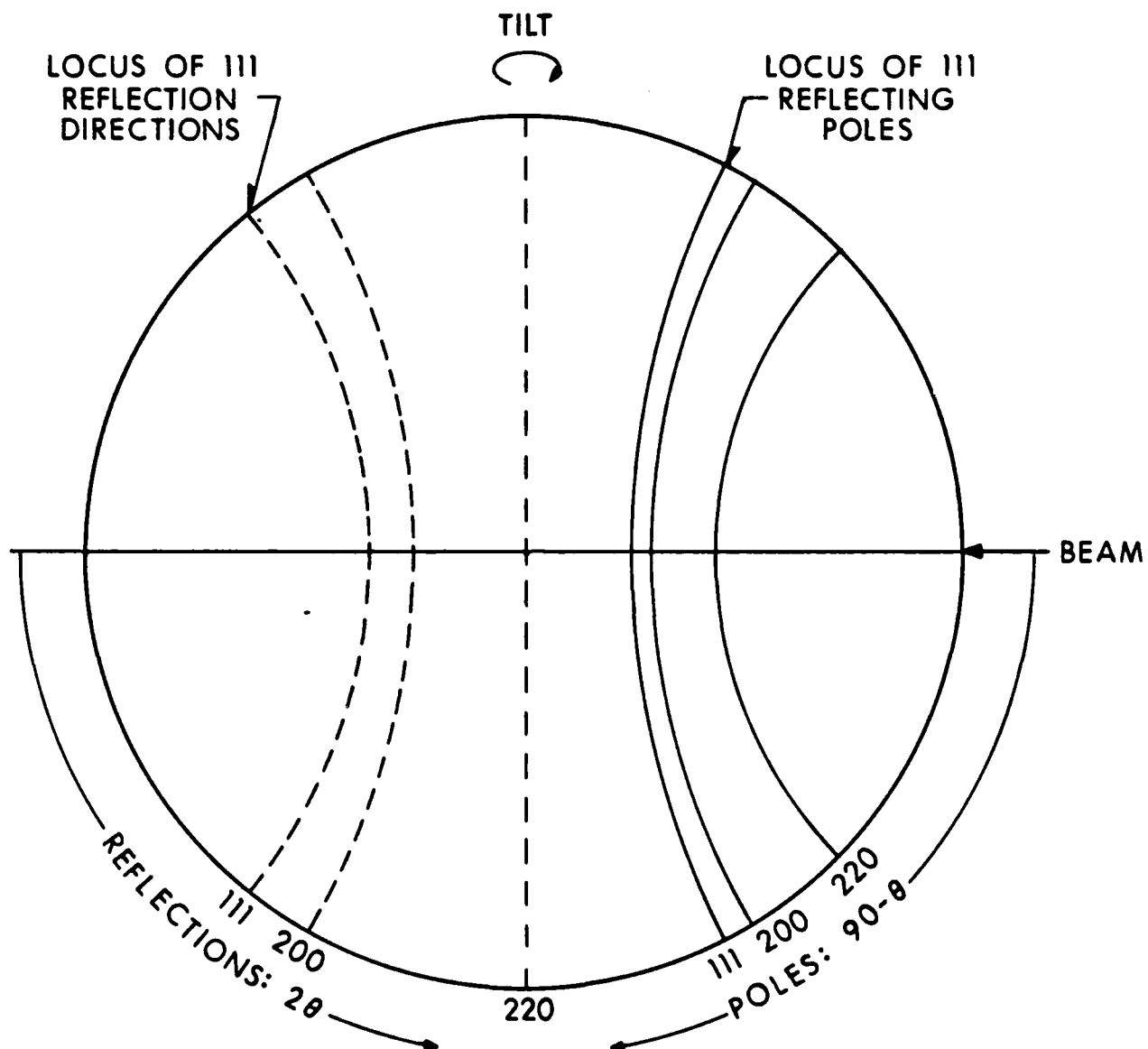Figure 1: Diffraction Geometry for Back Reflection Topography; Copper Crystal with Cu $K_\alpha$ Radiation.
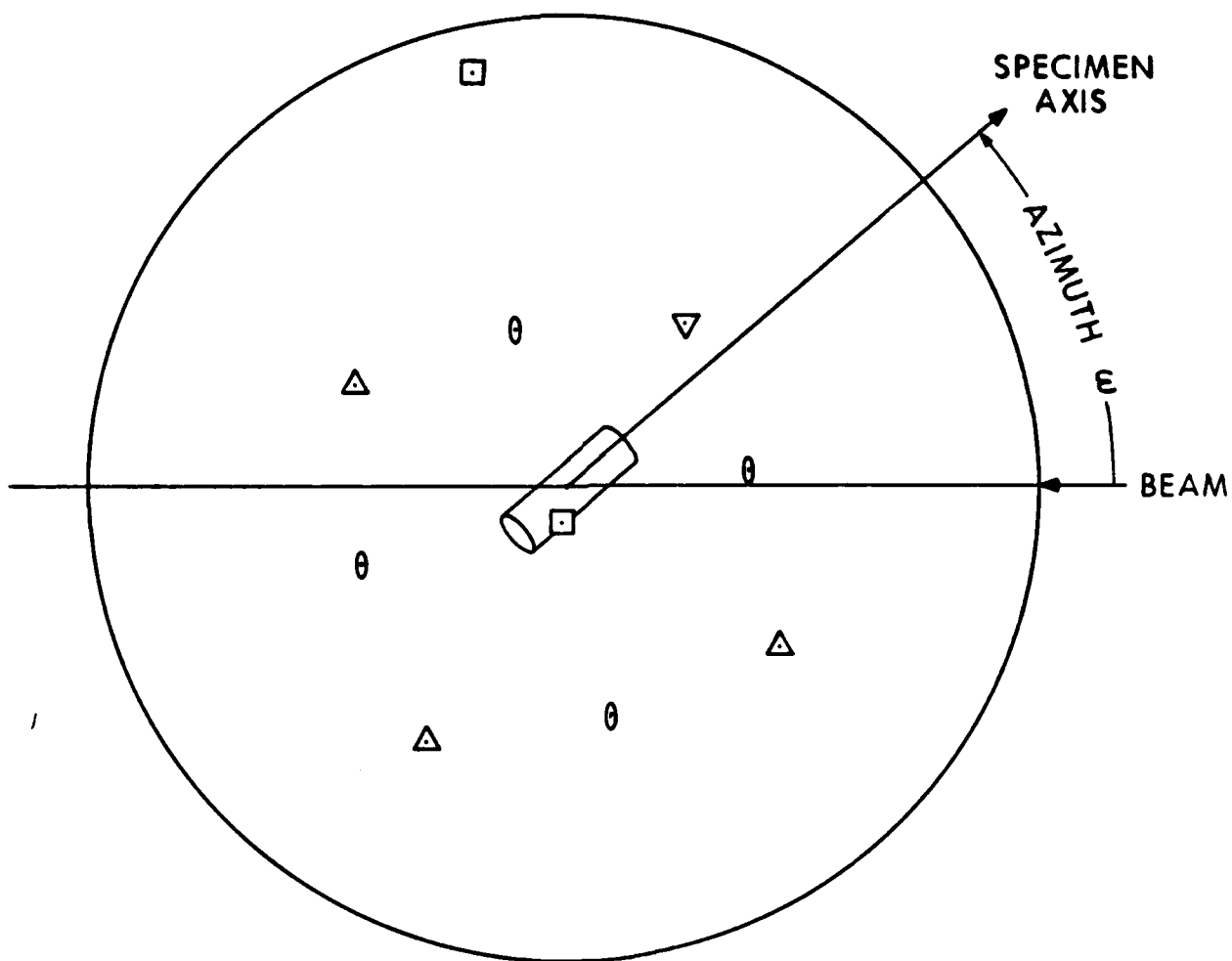
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Figure 2:   Stereogram of Crystal Prepared for Diffraction Analysis.
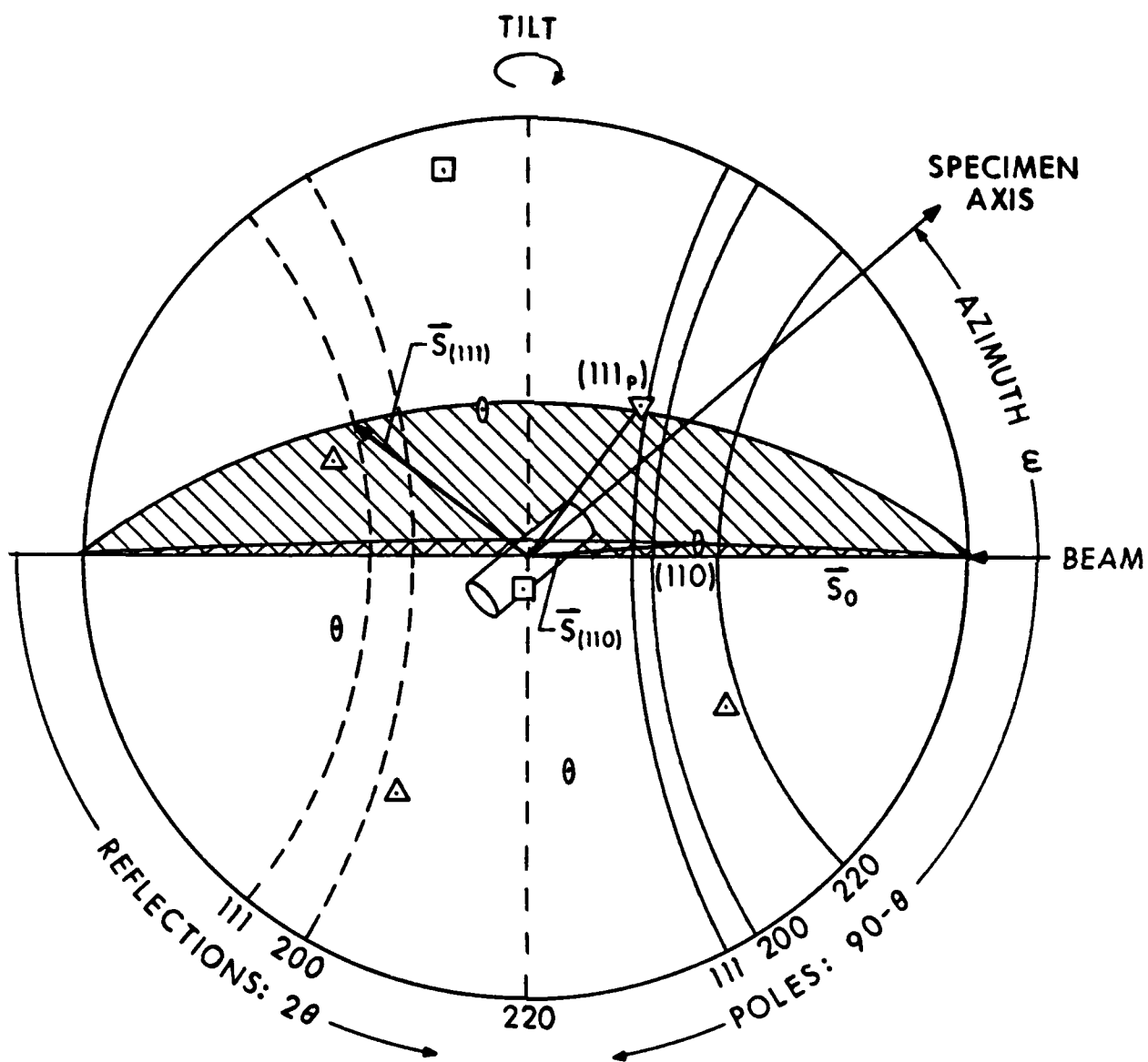
Figure 3:  Stereographic Analysis for Non-equatorial Reflections.

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just to the left of the reflecting locus, which could then be tilted a few degrees toward the incoming beam to achieve the Bragg condition.

Figure 2 is a stereogram of a copper crystal having a flat rectangular observation surface (sketched at the center) a few degrees from (001). The projection shows the normal to the observation surface at the center and the reference axis at some azimuthal angle ω to the beam.

In Figure 3 these plots are superimposed. The interpretation of the combined superimposed plots is now as follows:

Any plane whose pole touches its corresponding "locus of poles" about the incoming beam will be in diffracting position. Any pole lying to the left of its "locus of poles" can be brought to reflecting position by rotating the stereogram about the "TILT" axis (corresponding to tilting the sample into the beam) OR by rotating the stereogram about its center (corresponding to azimuthal rotation of the sample). The small circles on the left represent the "loci of diffracted beams" for (001), (011) and (111) respectively. When the pole of a diffracting plane touches its circle of poles, the plane of incidence can be represented by a great circle through the direct beam (at the equator) and the pole. The inter-section of this great circle with the corresponding locus of reflections defines the direction of the diffracted beam.

For the example shown, a (220) equatorial reflection is seen to be possible with a tilt of several degrees. The diffracted beam will emerge almost normal to the specimen surface. However, since there is also a (111) pole near its reflecting circle, it is likely that there will be simultaneous reflection, and over-lapping images. It can also be seen that by clockwise azimuthal adjustment, the (111) pole can be brought closer to reflection while the (220) moves farther away; thus it is probable that a grazing incidence (111) reflection could be obtained without interference from the (220). Finally, it is seen that by a further azimuth rotation a second grazing-incidence reflection setting is possible (this is in fact the case for any plane which lies more than θ degrees from the observation surface and thus cannot reflect in equatorial geometry). This property of non-equatorial reflections not only improves the probability of finding a setting free from overlapping images, but also opens up possibilities for contrast experiments, since it is possible to observe contrast changes in particular features even when the overall image quality may be poor. It is also seen from this plot that even poles lying close to the observation surface - such as the (010) pole shown near the north pole -- will also be in diffracting position at some setting. Although the diffracted beam emerges close to the surface and such reflections are not generally useful for topography, their presence as simultaneous reflections can be bother-some because of the small film-to-specimen distance.

The construction of the locus of poles and the locus of reflections was originally proposed by Leber to predict the range of reflections theoretically possible for a single-axis camera by specimen tilt alone. From the foregoing discussion, however, it is seen that when used in con-junction with the sample stereogram to describe a goniometer with an addi-tional 360° azimuth rotation it becomes a powerful tool for analyzing the complete specimen diffraction geometry, optimizing sample settings, and

devising appropriate strategies for contrast experiments and sample manipulation. The analysis can be further extended by the use of additional charts constructed for other radiations if available. For example, an interference between two desired reflections using copper radiation may be eliminated by switching to cobalt. Still another use is in selecting planes for sectioning the sample. Before actually sectioning the sample, a stereogram can be prepared for a proposed section and the possible imaging strategy checked in advance.

## III. EXPERIMENTAL PROCEDURE

The theoretical analysis of the previous section is applied as standard practice to optimize the conditions for topography. A stereogram of the sample is prepared with the observation normal at the center, showing all the principal Bragg reflecting planes. A chart similar to Figure 1 for the specimen crystal and the appropriate radiation is prepared on a separate sheet of tracing paper, and both charts are then superimposed on a Wulff net. Setting the beam direction on the diffraction chart at the north pole and rotating the stereogram corresponds to azimuthal rotation of the sample in the beam. A great circle through the pole of the diffracting plane represents the plane of incidence, and its intersection with the locus of reflections for this plane predicts the angle of the diffracted beam. By suitable manipulations, appropriate reflections can be selected and approximate settings predicted. The criteria to be optimized are: (1) low tilt angle (maximum area irradiated, and minimum sample-to-film distance) (2) no interfering simultaneous reflections (3) diffracted beam emerging nearly normal to sample, or at least close to equator (minimizes image distortion).

The camera designed for this work is shown schematically in Figure 4. For maximum convenience and versatility the collimator, the sample goniometer, and the film holder were designed as three separate units which mount on a long track fixed to the X-ray table. In order to utilize the width of the Phillips line focus, the camera is designed for a vertical plane of incidence. The collimator is a 20 cm brass tube which accomodates a Phillips diffractometer slit at the exit. The film holder was designed to permit the use of several sizes and types of film and the film-to-sample distance is adjustable with a screw. The sample goniometer is shown schematically in Figure 5. Larger samples are mounted directly on the goniometer with wax; samples several centimeters across and a centimeter or more in thickness can be accommodated. Very small samples are mounted in a separate holder before mounting on the goniometer. A full 360° azimuth rotation is provided, as well as a full range of specimen tilt. (A third rotation allows additional specimen adjustment; this is not normally significant, but the additional rotation axis permits the goniometer to be easily converted to horizontal plane of incidence if required.)

With the sample mounted on the goniometer and set approximately, final adjustment is done in the beam using a fluorescent screen or image tube.

When a satisfactory topograph has been obtained, the diffraction conditions are verified using a double exposure technique. The standard film holder, as shown schematically in Figure 5, was designed to hold a small
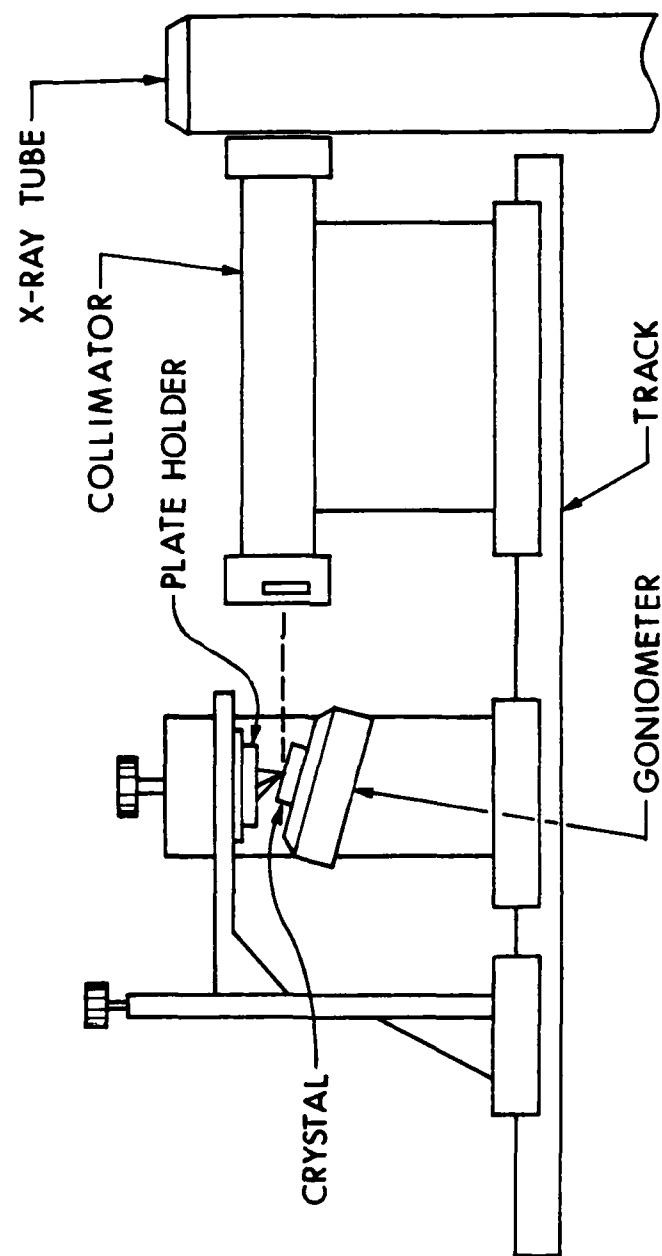
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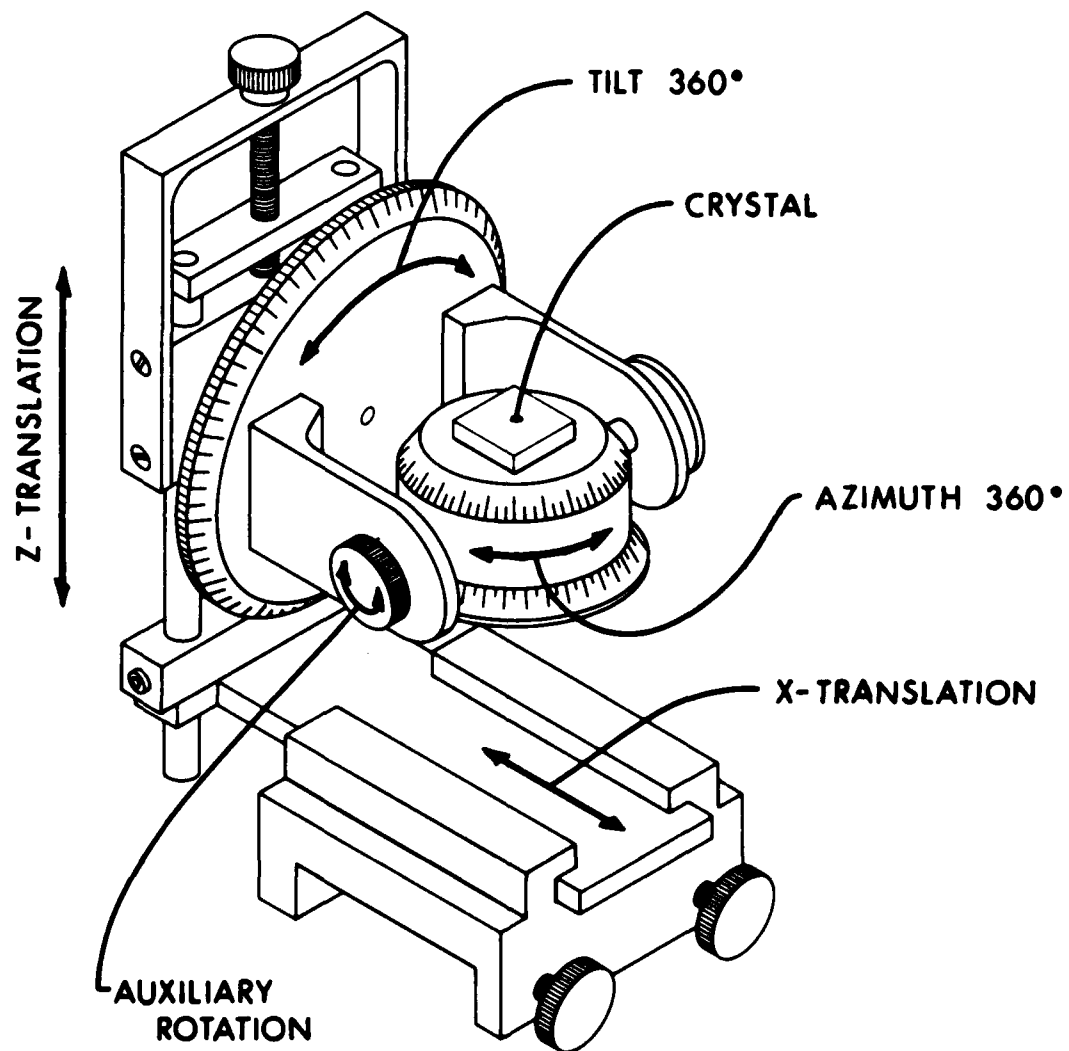Figure 4: Schematic Arrangement of Camera for X-ray Macrotopography.

Figure 5: Schematic of Sample Goniometer for X-ray Macrotopography.

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film or nuclear track plate in close proximity to the specimen surface. A second film holder unit was also designed to hold a 5" x 7" film at selected positions for the double exposure reflection check, as shown schematically in Figure 6.

Two exposures are made at different heights above the specimen. The identification of the images is readily apparent due to the poorer resolution and beam spreading with increasing distance. The presence of any unwanted reflection is also obvious, since the stray reflection will separate rapidly from the main image. The Bragg angle for the reflection is easily obtained by geometry. For equatorial reflections, both images will lie along the incident beam direction and the Bragg angle is determined from the image displacement $\Delta y$ and the film displacement $\Delta h$:

$$\tan 2\theta = \frac{\Delta h}{\Delta y}$$

For non-equatorial reflections the geometry is shown in Figure 7 where the incident beam $\vec{s}_0$ lies along the y-axis, the film is parallel to the xy plane, and the diffracted plane beam $\vec{s}_1$ lies in some arbitrary direction. The angle $2\theta$ can be determined from the dot product

$$\vec{s}_0 \cdot \vec{s}_1 = |\vec{s}_0| \, |\vec{s}_1| \cos 2\theta$$

where

$$\vec{s}_0 = \vec{j} \qquad\qquad\qquad |\vec{s}_0| \equiv 1$$

and

$$\vec{s}_1 = (x_2 - x_1)\,\vec{i} + (y_2 - y_1)\,\vec{j} + \Delta h\,\vec{k}$$

where $\Delta h$ is the film translation (Figure 6) and the x and y coordinates are measured from the film (Figure 8).

In some cases, this technique can also be used to check on the true nature of certain features, such as sub grains or very low angle boundaries, which are within the angular divergence of the oncoming beam. The nature of these features becomes apparent from the presence of image spreading as the distance from the sample increases.

Since X-ray topography does not introduce any magnification, the topographs are normally viewed with a binocular microscope. For measurement and analysis, the topographs can be backlighted on a light box and photographed with a copy camera. For higher magnifications or high quality prints, a two-step procedure was used in which the topograph was backlighted and photographed using a macro camera and fine-grain film, and then printed in an enlarger. As with any photographic process, the appropriate choice of films and final maginifcations is dependent upon the image resolution.
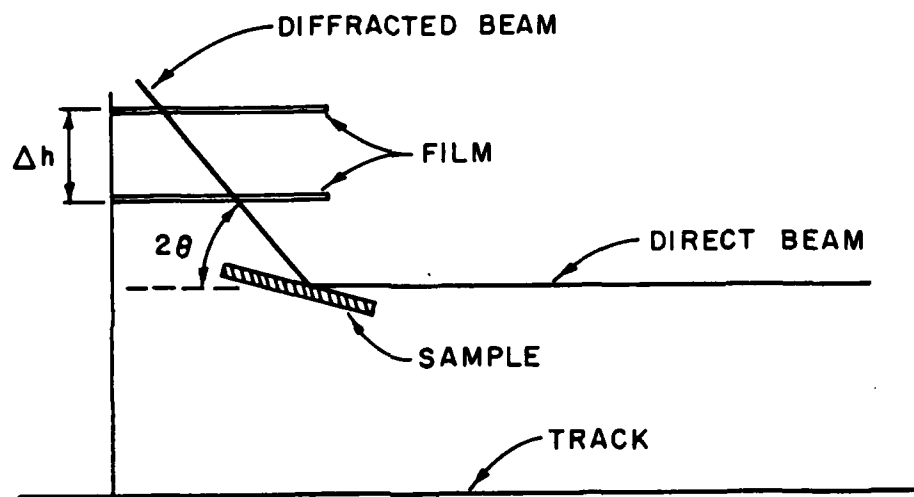
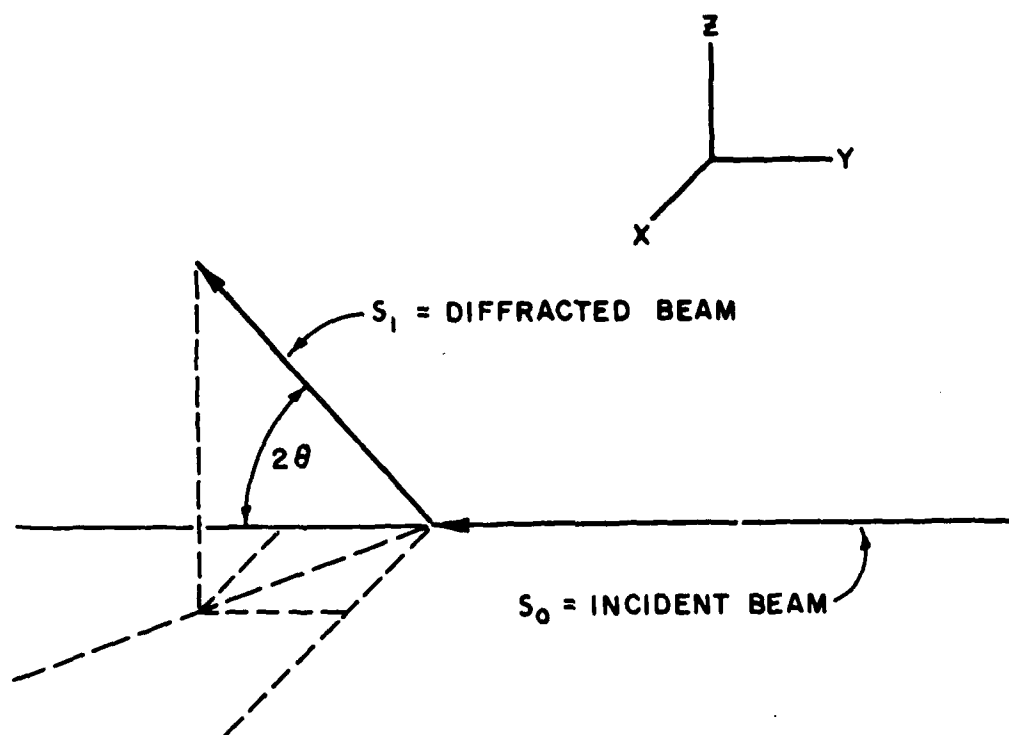Figure 6: Double Exposure Technique for Reflection Indexing.

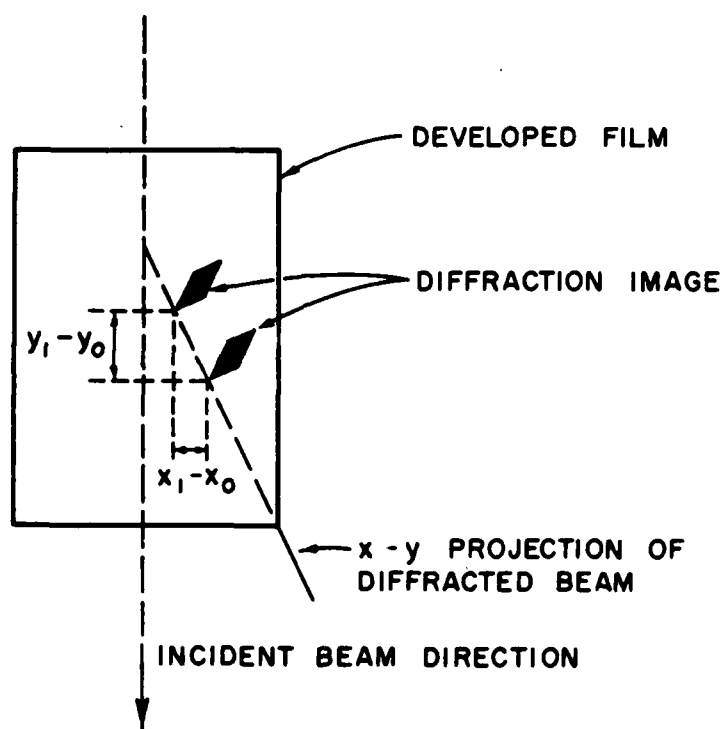Figure 7:  Geometry of Non-equatorial Reflections.

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Figure 8: Coordinates for Indexing Diffracted Image.

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# IV. SUMMARY

The principal thrust of X-ray topography has been toward the study of general dislocation arrangements in quite perfect crystals. Since the early work of Barrett and Honeycombe there has been relatively little interest in deformation structures; at least in part because of the difficulty of extracting useful quantitative information about the geometry from topographs. In this paper, the general geometry of back reflection topography has been completely described, and it has been shown how a systematic exploitation of the back reflection geometry can be used to maximize the information which can be obtained and optimize the diffraction geometry for any given Bragg reflection. A camera design based upon this general approach has been described, as well some specific procedures for its utilization; however, the most important feature is the general analysis of the back reflection technique. The basic approach to back reflection topography which derives from this analysis can be applied to any topographic arrangement. Since the present camera was intended for the study of macro-structures of crystals with high dislocation densities, it was designed to image large sample areas at low resolution. The basic analysis and procedures apply equally well, however, to any back reflection technique. The possibility of effectively utilizing non-equatorial reflections greatly enhances the power and flexibility of the method.

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6.  General Comments.  What do you think should be changed to improve future reports?  (Indicate changes to organization, technical content, format, etc.)

_____

_____

_____

CURRENT
ADDRESS

Name _____

Organization _____

Address _____

City, State, Zip

7.  If indicating a Change of Address or Address Correction, please provide the New or Correct Address in Block 6 above and the Old or Incorrect address below.

OLD
ADDRESS

Name _____

Organization _____

Address _____

City, State, Zip

(Remove this sheet along the perforation, fold as indicated, staple or tape closed, and mail.)

# END

# 6-81

# DTIC